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ACCOUNT

OF THE

ALUM-PROCESS

FOR

REMOVING POTASH, AMMONIA,

AND OTHER IMPURITIES,

FROM

SACCHARINE SOLUTIONS.

RECENTLY PATENTED

BY

JAMES DUNCAN, JOHN A. R. NEWLANDS,

AND

BENJAMIN E. R. NEWLANDS.

LONDON:
HENRY GILLMAN, BOY COURT, LUDGATE HILL, E.C.
1875.

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193. f. 55.

Printed at the Chemical News Office,
Boy Court, Ludgate Hill, London, E.C.

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ALUM-PROCESS FOR REMOVING POTASH,
&c.

THE following pages contain a brief account of the Alum-Process (recently patented by James Duncan, John A. R. Newlands, and Benjamin E. R. Newlands) for removing Potash, Ammonia, and other Impurities, from Saccharine Solutions.

In the manufacture of sugar from various sources, particularly from beet-root, syrups are formed containing large quantities of potash salts; and it is well known that the presence of these salts prevents the separation of a considerable portion of the sugar by the usual process of crystallisation.

On this point a few remarks may be quoted from various authorities who have written upon the subject of beet sugar.

Extract from a Paper by Dr. Wallace, of Glasgow. ("The Sugar Cane," August 2, 1869):—

"*Analysis of Salts from Beet Molasses.*

Chloride of potassium	18·70
Sulphate of potash	4·18
Carbonate of potash	53·80
Carbonate of soda	20·81
Carbonate of lime	0·35
Magnesia	0·27
Moisture and loss	1·89
	100·00

From these results it will be observed that the salts consist in great part of alkaline carbonates.

"In France, large quantities of carbonate of potash and other salts are obtained from beet molasses, by fermenting, distilling off the spirit, evaporating the spent wash, charring, and extracting the salts, which are afterwards separated by taking advantage of their different degrees of solubility.

"When beet sugars are used in the refinery, and especially under the Greenock system of turning out no syrup, the salts frequently accumulate in the liquors to an alarming extent, and render the system referred to a far from profitable one. Under such circumstances the syrup drained by the centrifugal machines from the low "yellows" occasionally contains from 4 to 5 per cent of alkaline salts; and if we accept the French calculation, which is, I believe, not far from the truth,—that every 1 per cent of these salts prevents the crystallisation of 5 per cent of cane sugar,—we can readily understand how it is that the working of these syrups is often very unsatisfactory. It is true that beet sugars contain a mere trace of fructose, but they introduce into the syrups an ingredient which is in some respects even more objectionable. The proportion of alkaline salts in beet sugars, such as are used by refiners in this country, varies from 1 to 4 per cent, and in extreme cases a larger quantity has been observed. Refiners would do well to reject all beet sugars that contain more than from 1½ to 2 per cent of salts."

Extract from the Work of Mr. Wm. Crookes, F.R.S. ("On the Manufacture of Beet-root Sugar," 1870, p. 121).

"The quantities by weight of potash, soda, and lime in the ash of beet-root molasses amount, respectively, to 51·72, 8, and 5 per cent, which exist mostly in combination with twenty-five parts of carbonic acid." And again (p. 250)—"The large quantity of potash in the ash of the beet-root soon attracted the attention of sugar manufacturers, and it was not long before attempts were made to utilise the beet residues for the preparation of potash salts. Boussingault found in two kinds of beet-root ash, respectively, 48·9 and 30·1 per cent of potash. Krocker found the ash of beet molasses to contain 49·6 of potash."

The following analyses, made in the laboratory of Mr. James Duncan, bear out the same view:—

Analysis of Lavenham Beet Syrup.

Sugar	60·49	
Ash	3·40	containing Potash 1·36
Water, &c.	36·11	
	100·00	

Analysis of French Beet Molasses.

Sugar	50·66	
Ash	10·86	containing Potash 4·88
Water, &c.	38·48	
	100·00	

The analysis of various samples of low cane sugar shows that they also contain a notable proportion of potash:—

	Dutch Bastards.	Guatemala.	Penang.	Low Penang.	Medium Penang.
Sugar ..	84·60	88·50	88·03	74·01	84·93
Glucose..	7·81	6·12	4·62	17·44	8·22
Potash ..	0·33	0·40	0·71	0·57	0·23

	Egyptian.	Egyptian.	Egyptian.	Jagghery.	Clayed Manilla.	Ila Ilo Manilla.
Sugar	83·76	86·00	85·43	78·85	84·54	82·73
Glucose	7·81	5·10	6·58	10·42	8·22	9·46
Potash	0·63	0·53	0·80	0·49	0·23	0·58

These analyses give the amount of potash present in the raw sugars, but the syrups resulting from such sugars by successive crystallisation would naturally contain a much larger percentage of potash.

The removal of the alkaline salts from saccharine solutions is therefore a question of the utmost importance; and on this subject Walkhoff, the great authority on beet-root sugar, writes in his treatise ("Du Sucre de Betteraves," vol. ii., p. 104, 1871) as follows:—"Efforts and researches ought to be directed towards the means of at *once eliminating* the alkalies from saccharine solutions, instead of seeking to transform them into combinations without deleterious action. The solution of this question will *completely renew* the face of the sugar industry, and will assure its future."

The potash present in saccharine solutions may be eliminated at once, and in the simplest possible manner, by means of the "alum-process," the exact nature and essential conditions of which will now be described. The process consists of two parts: 1st, precipitation of the potash in the form of alum; and 2nd, neutralisation of the residual acid liquor by means of lime.

1. *Precipitation.*—This is accomplished by adding to the cold syrup solution of sulphate of alumina, in quantity sufficient to form an alum with the whole of the potash present. It is convenient to work with syrup at a density of 38° B., and solution of sulphate of alumina at 27° B., or thereabouts. If the density of the syrup be much over 38° B. the alum cannot easily settle out. The mixture is well stirred for about fifteen minutes to one hour, and the whole allowed to repose for four or five hours, until the deposit—which consists of small crystals of alum, technically known as "alum meal" —has completely subsided. The tank in which this operation is performed is provided with mechanical stirring gear, and may be called the "alum-tank."

The three principal points to be attended to in this part of the process, in order to obtain the best results and to prevent the formation of glucose, are—

(1.) To work at the lowest attainable temperature;
(2.) To employ solutions as dense as possible; and
(3.) To perform the whole operation as quickly as possible, consistently with due separation of the alum.

The amount of potash present in syrups is generally equal to two-fifths of the ash. The ash is determined in the usual way, by addition of concentrated sulphuric acid, followed by incineration and weighing, one-tenth being deducted from its weight. It is sufficient, for most practical purposes, to assume that two-fifths of the ash is potash.

Every 1 part of potash requires for conversion into alum about 9½ parts of sulphate of alumina, out of which 2½ parts are required to convert the potash into sulphate, and the remaining 7 to combine with the sulphate of potash, so as to form alum. If the liquor contains any sulphuric acid, either free or combined, or if the solution of sulphate of alumina contains any free sulphuric acid, the 2½ parts of sulphate of alumina required to convert the potash into sulphate may be partly or entirely dispensed with.

For practical purposes it is sufficient to determine the percentage of ash, to assume two-fifths of this to be potash, then to multiply the percentage of potash by 9·5, which gives the dry sulphate of alumina, and, lastly, to ascertain the amount of solution corresponding to this by means of a table given further on.

2. *Neutralisation.*—The alum-tank is provided with several taps, at different heights, and, when the alum has well settled down, the clear acid liquor is run off, by means of these taps, into another tank placed on a lower level, and also provided with mechanical stirring-gear. This tank may be called the "limeing-tank." As soon as the acid liquor has been thus decanted into the limeing tank, a little finely-divided chalk, previously made into a paste with water, is added, so as to produce a slight effervescence. Milk of lime is then added at frequent intervals, until the froth has nearly, but not entirely, disappeared: the gradual abatement of the froth serves to indicate when the neutralisation is nearly complete. This operation takes from one to two hours. The point at which the neutralisation is practically complete may be known by three simple observations:—

(1.) The absence of any large amount of froth.

(2.) The absence of any taste of aluminous compounds.

(3.) The liquor should give only a *dull* red tinge to blue litmus paper.

When the neutralisation is thus practically complete, the treated liquor is subjected to the same routine as the ordinary solutions of sugar in a refinery; that is to say, it is heated in the blow-ups to 150° F. (but not to a higher temperature), then passed through Taylor filters, and through char, and, lastly, boiled down in the vacuum pan.

To wash and dry the precipitated alum, it is convenient to employ a small centrifugal machine. After once machining for a few minutes, a little water being added as usual during the operation, the alum appears white and dry, but still retains a small amount of syrup. It is then mixed up with some cold water, and machined a second time, after which it will be found free from sugar and fit for sale.

The following analyses show the effect of the process on Lavenham beet syrup, when treated on a large scale :—

	Lavenham Beet Syrup.	Ditto, after Treatment, and before Char.	Ditto, after Treatment, and after Char.
Sugar	60·18	40·54	41·60
Ash..	3·61	1·33	0·47
Water, &c. ..	36·21	58·13	57·93
	100·00	100·00	100·00

The advantages of the process are—

(1.) The removal of potash and ammonia from syrups without much dilution.

(2.) The removal of a great deal of the colouring and albuminous matters.

(3.) A considerable improvement both in taste and odour.

(4.) The alum produced is nearly equal in value to the sulphate of alumina used, so that the expense of the process is not great.

(5.) The plant required is of the simplest description, the cost of labour small, and the entire process is of a continuous and rapid character.

This process has been in constant operation during the past twelve months at the sugar-refinery of Mr. James Duncan, Clyde Wharf, Victoria Docks, London, where the syrup from many thousands of tons of sugar has been treated with excellent results, several hundred tons of potash alum, of good quality, being, during the same time, produced, and sold at a fair market-price. Licenses to work the process have been taken out by nine of the principal refiners of the United Kingdom, and licenses have also been granted to several other refiners in Holland, Belgium, and the United States of America.

The foregoing description applies to the alum-process as actually conducted at the Clyde Wharf Refinery: there are, however, various alterations which suggest themselves as likely to be of advantage under particular circumstances; and some of these will now be given by way of appendix, together with remarks on the different stages of the process.

APPENDIX.

Extent to which Potash is Removed by the Process.

Numerous experiments upon the large scale have conclusively shown that by means of the alum-process potash may be almost entirely separated from syrups, not more than 0·25 per cent being left in solution. After removing some of the sugar by crystallisation the residual potash accumulates in the syrup, and by treating this again with the alum-process the whole of the potash may be practically removed.

Mode of Using the Process so as to avoid Dilution.

Instead of using a solution, the dry sulphate of alumina, in a finely-ground state (or merely made into a paste with a little water), may be added to a syrup, and when alum separates under these circumstances the liquor is actually concentrated to some extent.

In operating directly upon beet sugars, they may be dissolved with agitation in a cold solution of sulphate of alumina, containing just sufficient to precipitate the potash.

Amount of Ash best suited for the Process.

It is not advisable to operate upon liquors containing less than 1 per cent of potash, as the alum in such cases, being small in amount, comes down rather slowly.

With liquors containing less than 1 per cent of potash, it is better to remove a portion of the sugar by the usual process of refining, and then to treat the residual syrup by the alum-process.

The syrups which are best adapted for the alum-process contain from 3·5 to 6·5 per cent of ash.

Composition of Sulphate of Alumina.

		Per cent.
1 equivalent of alumina =	51·4	15·41
3 equivalents of sulphuric acid =	120·0	35·99
18 ,, of water =	162·0	48·60
	333·4	100·00

It should be as free as possible from iron, and should not contain any large excess of sulphuric acid over that given in the analysis.

Sulphate of alumina is made by acting with sulphuric acid upon China clay, or kaolin, a silicate of alumina largely found in Cornwall, and elsewhere. The substance first obtained is a porous mass called alum-cake, consisting of sulphate of alumina mixed with about 20 per cent of silica. The alum-cake is dissolved in water, and the silica allowed to settle out, when the clear solution is evaporated down, and forms the solid sulphate of alumina. If alum-cake is employed, it is necessary to have tanks in which to dissolve it up and allow the silica to subside, when the clear solution is employed for the alum-process. It is not advisable to use the solution of alum-cake without first allowing the silica to settle out, as some of the latter comes down with the alum and reduces its value, whilst the greater part passes over into the neutralising tank, and renders the subsequent filtration more difficult. Alum-cake is cheaper than the ordinary sulphate of alumina; the price of alum-cake delivered in London being about £5 per ton, and that of sulphate of alumina about £7.

When the clear solution of alum-cake is used for the process, the whole cost of evaporating the sulphate of alumina to dryness is saved, it being at once precipitated, in the solid form, as potash-alum.

Table showing the Percentage of Sulphate of Alumina in Solutions of different Densities.

Baumé.	Sp. Gr.	Per Cent.	Baumé.	Sp. Gr.	Per Cent.
22½	1·184	34	28	1·225	43
24	1·188	35	29	1·235	44
24½	1·190	36	30	1·245	45
25	1·197	37	31	1·256	46
25½	1·201	38	31½	1·261	47
26	1·206	39	32	1·267	48
26½	1·211	40	32½	1·277	49
27	1·216	41	34	1·288	50
27½	1·218	42			

Composition of Potash-Alum.

		Per Cent.
1 equivalent of potash	= 47·0	9·91
1 „ alumina	= 51·4	10·83
4 equivalents of sulphuric acid	= 160·0	33·73
24 „ water	= 216·0	45·53
	474·4	100·00

Amount of Alum Produced.

When working with sulphate of alumina alone, the yield of alum is equal to about three-fourths of the sulphate of alumina used. When, however, a mixture of sulphate of alumina with sufficient sulphuric acid to sulphate the potash is employed, the yield of alum is about equal to the sulphate of alumina used.

Remarks on the Theory of the Alum-Process.

The effect of adding sulphate of alumina in the cold to a syrup containing potash salts is as follows:—

Syrup before Treatment—	Ditto, after addition of Sulphate of Alumina.	Ditto, after Neutralisation with Lime.
Contains potassium, combined with chlorine, nitric acid, carbonic acid, and various organic acids—that is to say, potash-salts.	The same acids (except the carbonic acid which escapes) in combination with aluminium—that is to say, alumina-salts.	The same acids (except the carbonic acid) in combination with calcium—that is to say, lime-salts, many of which are insoluble in syrup.

Now, it is true that owing to the equivalent of calcium 20, being about one-half that of potassium 39, the mere replacement of the potassium by calcium would lessen the ash; thus—

100 parts chloride of potassium = 74 parts chloride of calcium.
100 „ nitrate of potash = 81 „ nitrate of lime.
100 „ acetate of potash = 81 „ acetate of lime.

When beet syrups contain, as they almost invariably do, a considerable amount of carbonate of potash, the whole of this is practically removed, the carbonic acid being liberated on making the syrup acid with sulphate of alumina, and the potash being precipitated as alum. Many, also, of the organic acids form lime-salts which are insoluble in syrup, and this is well shown by the slight increase in the amount of lime after treatment, as compared with what might be expected to be present as the equivalent of the potash removed. Thus an experiment on the large scale with beet syrup, from Lavenham, gave the following results:—

	Before Treatment.	After Treatment.
	Per Cent.	Per Cent.
Sugar	60·00	46·00
Potash	1·36	0·25
Lime	0·11	0·21
Sulphuric acid	0·14	0·11

And calculating out the latter figures to correspond with the original amount of sugar, viz., 60 per cent, we have as follows:—

	Before Treatment.	After Treatment.
	Per Cent.	Per Cent.
Sugar	60·00	60·00
Potash	1·36	0·33—or 1·03 reduction.
Lime	0·11	0·27—or 0·16 increase.
Sulphuric acid	0·14	0·14—unaltered.

Now, it would require 0·61 of lime to replace the 1·03 of potash removed; and as the increase of lime is only about one-quarter of this, it is evident that, in this experiment, about three-quarters of the lime-salts produced were precipitated in the insoluble condition.

The alum-process, therefore, replaces the potash-salts by lime-salts, many of which are insoluble in syrup, and a good deal of what does dissolve is easily removed by animal charcoal.

The above remarks apply to the alum-process as usually conducted, when sulphate of alumina alone is employed; but it is possible, as stated in the same patent, to mix with the sulphate of alumina enough sulphuric acid to convert the whole of the potash into sulphate, and in this case the action of the alum-process is as follows :—

Before Treatment.	After addition of Sulphate of Alumina mixed with Sulphuric Acid.	After neutralising with Lime.
Potash-salts.	The free acids, with the exception of carbonic acid, which escapes.	Lime-salts, many of which are insoluble in syrup.

This mode of using the alum-process may appear more direct than the previous one : there are, however, two reasons which render it desirable to employ the sulphate of alumina by itself :—

1. Because the alumina salts are less likely to invert the sugar than the corresponding free acids.
2. The large precipitate of alumina produced helps to improve the colour of the syrup.

On the other hand, in favour of using a mixture of sulphate of alumina with sulphuric acid, the following advantages may be adduced :—

1. A saving of one-quarter of all the sulphate of alumina, the proportional yield of alum, as compared with the sulphate used, being therefore considerably greater.
2. As there are no alumina-salts left in solution (except the small percentage of alum which always exists in the syrup before adding lime) the amount of precipitate produced on neutralisation is but small, and is more easy to remove, either by filtration or otherwise.

Time required for Precipitation of Alum.

When solution of sulphate of alumina is mixed with a syrup containing potash, alum is precipitated almost immediately, and a very large proportion (sometimes as much as 95 per cent of the total alum) falls to the bottom of the tank within the first hour. The extra time allowed in working the process is simply for the purpose of enabling the finely divided particles of alum to precipitate themselves in a more complete manner. Much of this time may be saved by allowing an hour's rest, to enable the bulk of the alum to deposit, and then driving the supernatant liquor through a rough flannel filter, either by the application of atmospheric pressure or by a partial exhaustion, preferably by the latter.

Mode of Disposing of the Washings of the Alum.

The washings of the precipitated alum may either be returned to the next batch of syrup about to be treated, so as to re-deposit any small quantity of alum which may have passed through the machine, or they may be allowed to flow into the limeing-tank, and be at once neutralised therein.

On the Use of Chalk or Whiting to Neutralise the Acid Liquor.

After the separation of the alum it is possible to neutralise the acid liquor with chalk only, and this has been done on the large scale for a considerable time; and the use of chalk has one advantage over lime, that, should an excess be added, it does no harm to the syrup beyond simply increasing the insoluble deposit in the filters. There are, however, several disadvantages attending the substitution of chalk for lime, among which are the following:—The effervescence is very great; the chalk has to be added very gradually, to prevent frothing over; and the neutralising-tank must only be half filled. It is necessary occasionally to employ a jet of steam, or to throw in some milk of lime, so as to prevent the froth from flowing over the side of the tank. The mixing has to be continued for a long time, on account of the persistency with which the bubbles of gas remain imprisoned in the syrup, which must not be heated to expel them, being still more or less acid. For these and other reasons it is best to neutralise with lime, using only a little chalk.

On the Use of other Bases instead of Lime to Neutralise the Acid Liquor.

After the separation of the alum it is possible to neutralise the acid liquor with some other alkaline body instead of lime. Among other substances which have been tried for this purpose are—Ammonia, carbonate of ammonia; baryta, carbonate of baryta; strontia, carbonate of strontia; magnesia, carbonate of magnesia; but none of these substances give as good results as lime.

Separation of Lime from the Treated Liquor.

It has been already explained that the amount of lime in the treated liquor is far less than is equivalent to the potash replaced, because many of the lime-salts are insoluble in syrup. The remaining lime-salts are almost entirely removed by animal charcoal; but should it be thought more desirable to employ a chemical treatment, any lime may be easily removed by means of oxalate of ammonia, carbonate of ammonia, or phosphate of ammonia.

Separation of the Precipitate Formed on Neutralising with Lime.

This bulky precipitate consists almost entirely of alumina and sulphate of lime. It cannot be conveniently separated by an ordinary filter-press, because of the density of the liquor, say 27° to 30° Baumé. It is therefore necessary to employ Taylor filters for this purpose. If, however, the syrup be allowed to stand, or if the density be reduced to 20° B., or lower, a large part of the deposit will settle out, and then a filter-press may be advantageously used. When the treated liquor has been somewhat diluted it is possible to employ shallow cloth-filters, and to drive the clear liquor through these, either by the application of atmospheric pressure or exhaustion, preferably by the latter. After the deposit has become nearly free from liquor it should be well mixed with hot water, and the exhaustion again applied, and so on until all the sweet is removed.

Utilisation of the Precipitate Formed on Neutralising with Lime.

This deposit will, if acted upon by sulphuric acid, yield a large amount of sulphate of alumina, which can easily be separated, by filtration or decantation, from the comparatively insoluble sulphate of lime. Should the deposit contain much organic matter, it may be dried and gently heated, to get rid of this. Or the sulphate of alumina may be heated with some strong sulphuric acid, which will also destroy any organic matters present.

Mode of using the same Alumina continuously.

Under ordinary circumstances it is cheaper to buy sulphate of alumina, and to sell the alum resulting therefrom. Still cases may occur in which it may be difficult either to purchase sulphate of alumina or to dispose of the alum. If, however, the alum, once obtained, is dissolved in hot water, and lime added, a precipitate is formed consisting of alumina and sulphate of lime. This precipitate is then well washed with hot water, the washings, together with the original liquid, being evaporated till the sulphate of potash separates out on cooling. After the sulphate of potash has been washed out, the precipitate of alumina and sulphate of lime is heated with sulphuric acid, by means of which the sulphate of alumina is once more formed, and it can be readily separated, by filtration or decantation, from the almost insoluble sulphate of lime. In this way the alum-process can be continuously carried on, without either the purchase of any fresh sulphate of alumina (beyond a little to supply a slight accidental waste) or the permanent production of any alum. The only materials required are lime and sulphuric acid, the products being sulphate of potash and sulphate of lime.

Instead of decomposing the alum by lime or chalk other alkaline substances may be used, such as potash, soda, ammonia, baryta, strontia, or magnesia, or any of their carbonates.

If ammonia is used to decompose a hot solution of alum, the alumina, when thoroughly washed, with the aid of occasional pressure, should be dissolved up in sulphuric acid, so as to once more form sulphate of alumina. The liquid remaining after the separation of the alumina will, if evaporated down, leave a residue of sulphate of potash and sulphate of ammonia, which may be separated by heating. Or, during the evaporation, sufficient lime may be added to expel the whole of the ammonia, which can then be passed into a fresh quantity of alum solution. Thus the same quantity of ammonia (with slight additions, from time to time, to replace unavoidable loss) will serve to precipitate the alumina from any given amount of alum, the only products in this case being sulphate of potash and sulphate of lime.

Caution against Entering the Tanks.

Owing to the presence of carbonic acid gas in the limeing-tank no one should be permitted, on any account, to enter the same, as this gas will, as is well known, destroy life. A certain amount of carbonic acid gas may also be present in the alum-tank; and therefore, before any one descends into it to scrape out the deposited alum, the sliding-valve at the bottom should remain open for some minutes, so as to allow the carbonic acid gas to flow out, and thus remove any danger. In all cases it is advisable to have the tanks railed over, and the entrance locked, the key being placed in charge of a responsible foreman.

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